Chapter 6
Characterization Methods

6-1. Introduction

The most important activity prior to treatment is the proper characterization of site waste. Chemical characterization is covered in many other documents. The following is a brief summary of radioactive waste characterization issues. Proper characterization of the waste will directly influence the choice of treatment method. It is also important for proper design of a health and safety plan for the site. Field and laboratory methods used to identify and quantify concentrations of radionuclides in the environment are, in many cases, more exact, less costly, and more easily implemented than those employed for chemical analyses. Tables 6-1, 6-2, and 6-3 are taken from *Risk Assessment Guidance for Superfund,* Chapter 10, "Radiation Risk Assessment Guidance" (EPA 199 1). Tables 6-1 and 6-2 list types of laboratory and field radiation detection instruments. Table 6-3 gives lower limits of detection of selected radionuclides. Radioisotopic analytical detection limits are given in Table 6-4.

6-2. Measurement of Beta Radiation

a. Introduction. One key feature in the design of beta measurement techniques that differs from alpha or gamma measurement techniques is that beta particles emerge from nuclear decay with a spectrum of energies characteristic of each nuclide. This causes difficulties in isotope identification. Absolute isotope identification relies upon either spectral measurements or upon chemical separation techniques. Detection and quantification of the total beta radiation are important for radiation protection and in gross beta measurements required for the RI/FS process. Detectors may be designed for the measurement of particle flux (particles/cm^2-see) or for the measurement of dose equivalent (rem or Sv). In some instances, it may be easier or more convenient to use gammas associated with alpha or beta emissions or their daughters to measure the concentrations of the alpha- or beta-emitting radionuclides.

b. Gross beta measurements in water. Measurements of gross beta in water are standardized. The standard technique is described in *Standard Methods for the Examination of Water and Wastewater* (American Public Health Association 1976). The standard method deposits

Table 6-1
Types of Laboratory Radiation Detection Instruments

Type of Instrument	Typical Activity Range (mCi)	Typical Sample Form	Data Acquisition and Display
Gas Proportional Counters	10^{-7} to 10^{-3}	Film disc mount, gas	Ratemeter or scaler
Liquid-Scintillation Counters	10^{-7} to 10^{-3}	Up to 20 mi of liquid gel	Accessories for background subtraction, quench correction, internal standard, sample comparison
Nal(Tl) Cylindrical or Well Crystals	10^{-4} to 10^{-3}	Liquids, solid, or contained gas, <4 mi	Ratemeter Discrimination for measuring various energy regions Multichannel analyzer, or computer plus analog-to-digital converter Computational accessories for full-energy-peak identification, quantification, and spectrum stripping
Ionization Chambers	10^{-2} to 10^{-3}	Liquids, solid, or contained gas (can be large in size)	Ionization-current measurement; digital (mCi) readout, as in dose calibrators
Solid-State Detectors	10^{-2} to 10	Various	Multichannel analyzer or computer with various readout options

Source: EPA (1991)

Table 6-2
Types of Field Radiation Detection Instruments

Instruments	Range of Counting Rate and Other Characteristics	Typical Uses	Remarks
Beta-gamma surface monitors[1] Portable count rate meter	0-1,000; 0-10,000; 0-100,000 count/rein	Surfaces, hands, clothing battery powered	Simple, reliable, (thin walled or thin window G-H counter)
Alpha surface monitors		Surfaces, hands, clothing	Not accurate in high humidity;
Portable air proportional counter with probe	0-100,000 count/rein over 100 cm^2		battery powered; fragile window
Portable gas flow counter with probe	0-100,000 count/rein over 100 cm^2	Surfaces, hands, clothing	Not affected by the humidity; battery powered; fragile window
Portable scintillation counter with probe	0-100,000 count/rein over 100 cm^2	Surfaces, hands, clothing	Not affected by the humidity; battery powered; fragile window
Air monitors			
Particle samplers			
Filter paper (high volume)	400 ft^3/min (1.1 m^3/min)	For quick grab samples	Used intermittently; requires separate counter
Filter paper (low volume)	0.1 to 10 ft^3/min (0.003-0.3 m^3/min)	For continuous room air breathing zone monitoring	Used continuously; requires separate counter
Electrostatic precipitator	3 ft^3/min (0.09 m^3/min)	For continuous monitoring	Sample deposited on cylindrical shell; requires separate counter
Impinger	20 to 40 ft^3/min (0.6-1.1 m^3/min)	Alpha contamination	Special uses; requires separate counter
Tritium monitors	0.10 pCi/m^3/min	Continuous monitoring	May be sensitive to other
Flow ionization chambers			sources of ionization

[1] None of these surface monitors is suitable for tritium detection. Source: EPA 1991

the sample on a counting pan by oven or hot-plate drying. Counting is carried out with an internal gas proportional counter. This method is used as a screening technique throughout the United States. If both alpha and beta particle activity are present, they can be separated by measuring the counting rate versus voltage. Two plateaus will be present with the first due to alpha radiation only and the second due to alpha plus beta radiation.

c. Gross beta measurements in air. Air particulate collected on filter paper are counted with either end-window Geiger Muller (GM) tube, thin-window proportional flow counter, scintillation counter, or solid-state detector. The choice of detector depends primarily on whether concurrent gross alpha measurements are desired. In that case, an internal gas proportional counter would probably be chosen. External counting devices can be used to include or exclude alpha particles by varying the window thickness. Because beta spectra are so broad and because of the possible energy-dependent response of many counting instruments, it is desirable to calibrate with at least two or three beta radiation sources of differing energies.

d. Chemical separation techniques. There are many chemical separation techniques. In lieu of separation, one can measure a more easily identified daughter element to determine the concentration of a parent element. Separation may not be necessary when spectroscopic techniques can be used. If the element is a beta/gamma emitter, then gamma spectroscopy can be used. Electron spectroscopy can also be used to measure beta radiation emission. Germanium solid state detectors are utilized in electron spectroscopy.

6-3. Measurement of Alpha Radiation

a. Special considerations. The main difficulty encountered when trying to measure alpha radiation is the extremely short travel distance of the alpha particle. Because of this, detectors must have very thin windows, or the alpha particles are absorbed in the window and

Table 6-3
Lower Limits of Detection for Selected Radionuclides Using Standard Analytical Methods[1]

Isotope	Sample Media[2]	LLD pCi	LLD Bq	Methodology
Co-60	-Water	10	0.4	Gamma Spectrometry
	-Soil (dry wt.)	0.1	0.004	Gamma Spectrometry
	-Biota (wet dry)[3]	0.1	0.004	Gamma Spectrometry
	.Air[4]	25	0.9	Gamma Spectrometry
Sr-90	-Water	1	0.04	Radiochemistry
Ca-137	-Water	10	0.4	Gamma Spectrometry
		0.3	0.01	Radiochemistry
	-Soil (dry wt.)	1	0.04	Gamma Spectrometry
		0.3	0.01	Radiochemistry
	-Biota (wet wt.)	1	0.04	Gamma Spectrometry
		0.3	0.01	Radiochemistry
	-Air	30	1	Gamma Spectrometry
Pb-210	-Water	0.2	0.007	Radiochemistry
	-Soil (dry wt.)	0.2	0.007	Radiochemistry
	-Biota (wet wt.)	0.2	0.007	Radiochemistry
	-Air	5	0.2	Radiochemistry
Ra-226	-Water	100	4	Gamma Spectrometry
		0.1	0.004	Radiochemistry
		0.1	0.004	Radon Daughter Emanation
	-Soil (dry wt.)	0.1	0.004	Radon Daughter Emanation
	-Biota (wet wt.)	0.1	0.004	Radon Daughter Emanation
	-Air	1	0.04	Alpha Spectrometry
Th-232	-Water	0.02	0.0007	Alpha Spectrometry
	-Soil (dry wt.)	0.2	0.007	Radiochemistry
	-Biota (wet wt.)	0.02	0.0007	Alpha Spectrometry
	-Air	0.3	0.01	Alpha Proportional Counter
U-234	-Water	0.02	0.0007	Alpha Spectrometry
U-235	-Soil (dry wt.)	0.1	0.004	Alpha Spectrometry
U-238	-Biota (wet wt.)	0.01	0.0004	Alpha Spectrometry
	-Air	0.2	0.007	Alpha Spectrometry
Pu-238	-Water	0.02	0.0007	Alpha Spectrometry
Pu-239	-Soil (dry wt.)	0.1	0.004	Alpha Spectrometry
Pu-240	-Biota (wet wt.)	0.01	0.0004	Alpha Spectrometry
	-Air	0.2	0.007	Alpha Spectrometry

[1] Note that LLDs are radionuclide, media sample size, and laboratory specific higher and lower LLDs than those reported above are possible. The risk assessor should request and report the LLDs supplied by the laboratory performing the analyses.
[2] Nominal sample sizes: water (1 liter), soil (1 kg dry wt.), biota (1 kg wet wt.), and air (1 filter sample).
[3] Biota includes vegetation, fish, and meat.
[4] Air refers to a sample of 300 m^3 of air collected on a filter, which is analyzed for the radionuclide of interest. Source: EPA 1991

never make it to the detector. The same feature is a problem with thick samples. A thick sample will actually absorb the alphas being emitted from the far side of the sample, and the detector's reading will only indicate the radiation being emitted from the near side of the sample. Thus, care must be taken in sample preparation and in the choice of the detector window.

Table 6-4
Radioisotopic Analytical Detection Limits

Parameter	Matrix	Method	LIMS Code	Detection Limit
Gross alpha/beta	Water Soil	EPA 900.0/SW9310 SM 7110/SW9310	ABT/900.0/Q4 ABT5/SM7110/Q4	5 pCi/ℓ 25 pCi/g
Gross alpha	Water	EERF00.02/SW9310	RAD/EERF00.02/Q4	5 pCi/ℓ
Gamma scan	Water Soil	EPA 901.1 HASL 300	RAD/901.1/Q4 RAD/GAMMA/Q4	20 pCi/ℓ @ CS 137 2 pCi/ℓ @ CS 137
Radium 2261228	Water Soil	EPA 904.0 HASL 300	RAD/RA226228/Q4 RAD/GAMMA/Q4	2.5 pCi/ℓ 2 pCi/g
Total uranium	Water Soil	ASTM 5174-91 HASL 300	RAD/KPA/Q4 RAD/GAMMA/Q4	1 /ug/ℓ 10 pCi/g
Istopic U	Any	U-NAS-NS-3050	RAD/ISOU/Q4	1 pCi/matrix
Radium 226	Liq.	EPA 903.1	RAD/RA226E,AM/Q4	0.2 pCi/ℓ

b. Alpha spectroscopy. The most common alpha spectroscopic systems depend principally on semiconductor (solid state) detectors. Semiconductor detectors offer the advantages of low background noise, a high resolution capacity, and short analysis times. A silicon diode operated at room temperature is usually used for alpha spectroscopy. If the isotope is an alpha/gamma emitter, then gamma spectroscopy can be used for quantification.

c. Alpha particle measurements in water. Sample preparation is the difficult part of obtaining alpha measurements from water samples. Direct evaporation may result in a residue that absorbs some alpha particle energy and hence is not appropriate for high-resolution spectrometry. For gross alpha measurements, the water sample is dried by oven evaporation just below the boiling temperature, or on a hot plate, and then counted. The sample should be less than 3 mg/cm^2 in thickness. The main error in this procedure is the loss of particles which might become airborne during the drying process. The recovery of gross alpha activity in this procedure has been shown to be in the 85 to 90 percent range. For counting gross alpha radiation, an internal gas proportional counter is recommended. It has high sensitivity, good geometrical efficiency, and reliability. Alternative methods might employ either a thin-window proportional counter or a GM tube. The GM tube has the limitation of not separating alpha particle from beta particle activity.

d. Alpha particle measurements in air. Most alpha particles in air are in particulate form (radon is often described as an alpha particle; it is not, but its daughters are alpha emitters). Gross alpha particle activity in air can be measured by collecting an air sample on a filter and then using any of the detectors sensitive to alpha particles, such as gas proportional counters, scintillators, and semiconductor detectors. Radon and its daughters can be measured in a Lucas chamber, which is a small spherical or cylindrical shell lined with a zinc sulfide scintillator viewed by a photomultiplier tube. The sample, which may contain gaseous or particulate activity, is introduced into the volume and counted directly.

e. Chemical separation techniques. The short range of alpha particles often necessitates the use of chemical separation techniques to concentrate the alpha-particle emitter from the bulk material of the main sample. The initial sample may be water, soil, biological tissue, or an air filter. The final sample may be electroplated onto a metal planchet, precipitated onto a filter, or incorporated into a liquid scintillation sample. Most routine chemical separations employ acid leaching to remove the radionuclides of interest directly from the bulk sample.

6-4. Measurement of Gamma and X-Rays

a. Survey instruments. For absorbed dose measurements in the 150-keV to several-MeV region, the hand-held ionization chamber is the primary survey instrument. Other counters such as GM tubes, gas proportional counters, and scintilla ion counters have serious, energy-dependent sensitivity problems. For survey use that does not require absorbed dose measurements, these other counters can be used with the scintillation counter being the most common. For x- and gamma radiation below 150 keV, scintillation counters are the most useful, with GM counters also frequently used.

b. Gamma and x-ray spectrometry. A gamma ray spectrometer can be used as a laboratory device where samples are counted and also as a field device for performing in situ analysis. The key factors in the choice of instrumentation are efficiency, resolution, background, energy range, sample capability, and cost. Since environmental measurements must be made of species that are many times at very low concentrations, there is a premium on sensitivity. High resolution is required to distinguish a minor activity in the presence of larger activities. Scintillation counters employing a sodium iodide crystal with a thallium activator have been a mainstay of gamma ray spectrometry since 1948. Of main consideration in the choice of an NaI(Tl) system are the crystal size, geometry, and the type of electronic instrumentation. The type of sample and level of contamination dictate these parameters. Semiconductor detectors are stable, reliable, and have good energy resolution. The most commonly used semiconductor detectors are germanium detectors. Silicon detectors are used for gamma spectrometry at low energies. The germanium detectors must be operated at liquid nitrogen temperatures (77° K); however, the silicon detectors can be operated at room temperatures but are inferior in resolution. The germanium detector is not as efficient as the NaI detector but is capable of distinguishing between very closely spaced energies. It is recommended that NaI(Tl) detectors be used to measure one (or a few) radionuclides at very low concentrations and germanium detectors be used to characterize a sample with a large number of radionuclides present. Germanium detectors are much more expensive than NaI(Tl) systems.

6-5. Nonintrusive Drum Imaging Techniques

Drums with unknown waste components pose a special problem to site characterization. Considering the radioactive component of the waste, it is imprudent to perform intrusive sampling. Currently, real-time radiography is used to image the contents of drums. Real-time radiography has limited contrast resolution so that dense objects cannot be imaged and has no three-dimensional imaging capacity. Digital radiography (DR) and computed tomography (CT) are being developed as nonintrusive drum imaging techniques. DR and CT system costs are several times higher because they require sophisticated computers, very precise mechanical systems under computer control, and detectors with much higher dynamic range. CT images represent a cross section of the object without the overlapping of features seen in real-time radiography. DR has much higher contrast resolution because of the detectors used. DR and CT also have the drawback of slower inspection speed, but this is being addressed.